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AD-A214 656

NOVEL ION SOURCES AND THEIR APPLICATIONS

A. LA JOLLA INSTITUTE WORKSHOP

3-4 AUGUST 1981

BY

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WITH CONTRIBUTIONS FROM

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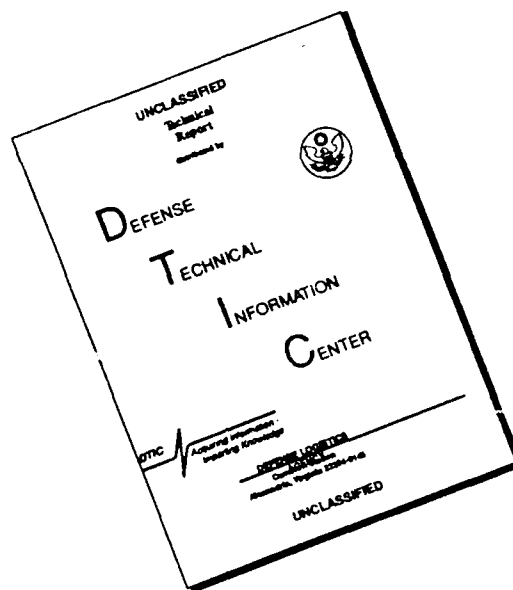
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EXECUTIVE SUMMARY

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A meeting was held at the La Jolla Institute on 3-4 August 1981, to have experts discuss recent research in high brightness ion sources with an eye toward possible applications of importance to DoD. Participation was invited from the National Submicron Facility at Cornell University, the Stevens Institute of Technology, the Brookhaven National Laboratory, the Oregon Graduate Center, the University of California at Los Angeles, the University of California at Irvine, United Kingdom Atomic Energy Authority Culham Laboratory, Phrasor Scientific, Inc., and Thermo Electron Corporation. Of these, the U.K.A.E.A. representative was unable to attend but provided us with a written account of his recent work on liquid metal ion sources. Dr. A. Maschke (Brookhaven National Laboratories) was also unable to attend, but indicated that he has reproduced the Phrasor Scientific results on the capillaritron using argon. He intends next to study the focusability of ion beams produced by capillaritrons.

A. T. FORRESTER (University of California, Los Angeles) presented an overview of the definitions of fundamental concepts used to characterize ion beams. He pointed out the usefulness of normalized perveance for comparing beams of different ions. The unit poissance was suggested for this parameter. Another quality which can be usefully normalized is the brightness. In terms of the normalized emittance, $\epsilon_N = \beta \gamma \epsilon$, the normalized brightness is defined by $B_N \equiv B^2 / \beta^2 \gamma^2 = \eta I / \pi^2 \epsilon_N^2$ which compares with the brightness per volt; η is a geometric factor of order unity.

A. T. FORRESTER AND J. PEREL (Phrasor Scientific, Inc.) summarized the work at Phrasor Scientific on capillaritrons in which a gas at high pressure (several hundred torr) is forced through a conducting capillary kept at a potential of about 10 kV with respect to an annular anode. Ions are produced from the plasma near the tip of the capillary by a process not

completely understood. Current densities are in the range of 100-1000 A/cm². A range of gases has been employed including helium, neon, argon and xenon. The source provides a very quiet beam with a relative fluctuation less than 0.1%. Drawbacks are: substantial energy spread (possibly due to charge exchange) which is comparable to the accelerating potential, and low gas efficiency (a few percent). However, for some gases, notably helium, there is a marked peak in the energy profile. Work is underway to isolate the dominant mechanism(s) for the energy spread to determine the inherent limits on an energy spread of this source. Dr. G. Hanson suggested that the energy spread due to charge exchange could be reduced by deflecting the neutral beam away from the ion beam.

Possible applications cited for the capillaritron are as a source for neutrals production; production of negative ions by directing the ions at a surface coated with cesium; ion implantation for surface hardening; and annealing. If the energy spread can be reduced this source could be useful in ion beam writing applications and lithography. Also, its use as a driver for the MEQALAC cannot be ruled out.

The work on droplet sources at Phrasor Scientific was also discussed. The source is a liquid metal ion source with the droplet size set by the extracting voltage and the current. Droplet sources have been used to study the process of rapid solidification with cooling rates up to 10⁵ °K/sec. As the work at U.K.A.E.A., Culham, indicates (see Appendix C), sub-um droplets can be produced by this method. If the particle size distribution can be made sufficiently narrow, novel applications may arise in tribology, abrasives, and applications where homogeneous packing of solids is important.

The study of cooling of bodies whose size is comparable to or smaller than the wavelengths of the emitted radiation is interesting in its own right. Does the Stefan-Boltzmann law apply here, for instance?

A. E. BELL (Oregon Graduate Center) discussed the recent work of his Group on Liquid Metal Ion (LMI) sources. In these sources, a metal is heated to its melting point and then wets a blunt needle of tip radius 5-10 um. Under the influence of an electric field the liquid metal is drawn into a cone whose extent is set by the balance between the electric field pressure and the surface tension stress. Ions are produced by field emission. For use in an LMI source, therefore, the

metal should have a low vapor pressure and high surface tension. It must also be able to wet the needle without corroding it. These constraints have in the past limited the use of these sources to gallium and gold. A widening variety of ions is now becoming available including: Ga^+ , In^+ , Bi^+ , Al^+ , Li^+ , Cs^+ and Au^+ . More recently, liquid alloys of platinum and boron and of platinum and arsenic have made boron and arsenic sources available. Other useful dopants now available from LMI sources are tin and silicon.

Corrosion of the needle (usually tungsten) has limited the lifetime of these sources, however. Bell and co-workers have found that a graphite needle substantially increases the lifetime of aluminum LMI sources and will probably also do so for sources of boron and arsenic ions. The difficulty of wetting and corrosion with aluminum has been overcome by first diffusing titanium into the graphite which is then covered by aluminum. Availability of long life boron and arsenic sources would render possible direct implantation of their ions.

Current liquid metal ion sources produce high angular intensities ($\sim 20 \mu\text{A}/\text{sr}$) and moderate energy spreads ($\sim 20 \text{ eV}$). But the present work on LMI sources as reported, for instance, at the 28th International Field Emission Symposium (Beaverton, 27-31 July 1981) indicates that these sources are constructed without much fundamental understanding of how they work. Unanswered questions are:

- i) What mechanism(s) are responsible for the energy spread?
- ii) What is the ionization mechanism?
- iii) Are the ions drawn from a (Taylor) cone or a whisker?
- iv) Is the emission space charge limited or is it set by hydrodynamic limitations (by flow rate or instability)?
- v) Can the wetting process be explained such that optimal needle composition can be predicted for any given metal?
- vi) What is the theoretical limit on the figure of merit (for fine focus applications)?
- vii) What is the process by which neutrals are formed and why are they under certain conditions emitted into a narrower cone than that for the ions?
- viii) What is the effect of the needle's surface condition on the current versus voltage curve?

On the last point, it appears from work at Bell Telephone Laboratories that a rough needle surface leads to greater emission for a given extraction voltage than a smooth surface.

On applications: J. H. Orloff has investigated the writing speeds of LMI sources for direct ion implantation fabrication of VLSI circuits and concluded that LMI sources at lower ion doping levels (10^{12} - $10^{13}/\text{cm}^2$) will be able to meet the VHSIC II goals at 0.5 μm resolution level. Higher dosing rates would be attainable if only small areas of the total wafer are doped. It is expected that direct write ion implantation will be the technology of choice in the future for applications requiring low levels of doping or moderately high production runs of VLSI circuits.

Other microfabrication uses of LMI ion beams include ion milling and resist exposure where ions have advantages over scanning electron systems because of greater resist sensitivity to ions and less degradation of line profiles due to charge particle scattering effects (proximity effects). A limitation to ion beam use in deflection systems is caused by relatively low ion speed which limits deflection rates to about an order of magnitude less than for electron beam systems. In addition to these uses, LMI sources will have application in SIMS—and for high density memory storage.

Non-fine-focused beam applications include surface modification to reduce wear and corrosion. A novel application for isotope separation can be envisaged at relatively low cost by using arrays of LMI sources; uranium might be a candidate for such a use.

For survey on liquid metal ion sources and developments in Japan, see Appendix A.

G. Hanson (National Submicron Facility) presented results on a gas phase field ion source developed recently at the National Submicron Facility. Since 1951, many researchers have developed an understanding of the physical processes which produce in a given field range point projection images of surface atoms. Using an understanding of the effects of neutral supply, temperature, electrostatic field, etc., a low temperature source has been developed which produces pure H_2^+ from a virtual point (few angstroms source size) with an energy spread of 1 eV fwhm (2 eV for 90% of ions) at an angular intensity of 20 $\mu\text{A}/\text{sr}$. This source is appropriate for very high resolution beam work: X-ray micro-analysis, H_2^+ ion resist lithography and radiation enhanced etch

processing at dimensions down to $\sim 100 \text{ \AA}$. The source brightness at 5 kV is calculated to be $\sim 10^3 \text{ A/cm}^2/\text{sr}$. With a good gun lens ($C_o = C_s = 2 \text{ cm}$) the effective brightness is $\sim 6 \times 10^7 \text{ A/cm}^2/\text{sr}$ (beam energy of 50 kV) where source current densities are $\sim 700 \text{ A/cm}^2$ (acceptance half-angle of 5 mr).

Control of the exact emitter configuration by microfabrication would allow the operation of such a source at much higher total current than in the present source (20 μA). This would permit its use for very large dimension ($0.1 \text{ }\mu\text{m} - 1 \text{ }\mu\text{m}$) beams. Microfabrication techniques are presently being investigated at the National Research and Resource Facility for Sub-micron Structures at Cornell (NRRFSS). Once control of the emitter surface configuration is achieved for the hydrogen source, the same principles could be applied to generate high optical quality beams of other species: dopants, metals, etc. The source intensity, species and energy spread would of course be a matter for investigation.

Further details are given in Appendix B.

M. SEIDL (Stevens Institute of Technology) presented an overview of his work on negative ion sources. Mechanisms used include:

- (a) thermionic emission (thermally activated field evaporation) from the surface of ionic conductors. Examples cited were O^- and O_2^- from zirconia (doped with CaO , Y_2O_3) and F^- from CaF_2 .
- (b) Sputtering of H^- ions from solids bombarded with Cs^+ ions. Substrates can be LiH_x , CsH_x , TiH_x , VM_x , Mo+ chemisorbed hydrogen, and Mo+ implanted hydrogen.

These have the advantage of small thermal spread and good gas efficiency. They also have the possibility of producing exotic ions.

The present status of the thermionic emission from ionic crystals is: Only ions with large electron affinity can be produced (e.g., halogens, oxygen). D.C. current densities of O^- and O_2^- up to 10 mA/cm^2 have been produced from ZrO_2 at 1200°C . This might be improved if a better understanding of the surface treatment needed were available. The current densities in short pulses are expected to be much larger.

On sputtering: Many ions can be produced. H^- beams have been obtained with a yield of 0.5 H^- per Cs^+ and an energy spread less than 3 eV.

Improvement of sputter sources may result from a better understanding of ion formation and from a detailed understanding of the processes causing the energy spread. Also needed are studies on the negative ions and the development of a pulsed surface plasma source.

The advantages of using negative ion beams result from the ease with which they can be neutralized without materially affecting the beam quality. The neutral beams can be used as the driver in inertial confinement fusion by ballistic focusing on the target and as exoatmospheric particle beam weapons. They are not subject to the usual electrostatic and electromagnetic instabilities and their compression is limited only by the thermal spread.

L. DANIELSON (Thermo Electron Corporation) reported on recent work on negative ion sources at Thermo Electron Corporation. Negative ion beams can be neutralized with high efficiency at high energies to produce intense neutral beams. Negative ion beams are formed from positive ion beams by volume production, double charge exchange, or surface production. Surface production appears to be most promising at this time. In this case, negative ions (e.g., H^-) are emitted from a low work function target immersed in a hydrogen-cesium plasma. The H^- ions are formed either by "reflection" of hydrogen ions from the surface or by sputtering by cesium ions.

The relative importance of reflection versus sputtering could be examined by supplying cesium to the back of a porous sample so sputtering by cesium is eliminated. Cesium diffusion through the target would maintain the required low work function.

Thermo Electron Corporation has developed a technique for preparing sublimed molybdenum samples. Conclusive experiments have shown that cesium can readily diffuse through these samples. These samples have also been doped with oxygen. These oxygen-containing samples may provide a lower work function in the presence of cesium than the bare metal samples presently employed.

Ultra high vacuum experiments including measurements of the work functions of cesium on sublimed molybdenum, diffusion rates of cesium through sublimed molybdenum, and desorption products are necessary to optimize the H^- yield from such a surface. Substantial improvements in H^- yield may be possible with the use of this novel cesium diffuser target.

P. D. PREWETT (U.K.A.E.A., Culham, U.K.) provided details on his work⁺ with field emission sources which are now commercially available through Dubilier Scientific Ltd., and the work on a closely related field emission system in which a mixture of metal ions and charged liquid droplets is produced by increasing the supply of liquid metal. A variety of metals can thus be sprayed onto substrates for the purpose of surface modification (hardening, etc.).

The work with gold deposited on glass showed that droplet size could be held to 2 μm . Coating rates were achieved of 0.5 - 1.0 $\mu\text{m cm}^2/\text{min}$ and the gold-glass adherence was measured to be $2 \times 10^7 \text{ N/m}^2$.

These sources can be used not only to produce highly adherent coatings but also for making electrical contact patterns, surface corrosion protection and metal bonding. Prewett points out that, because the ion beam is formed in high vacuum and is highly directional, it can be used to produce highly uniform patterned coatings with masks. At a current of 600 μA and an extraction voltage of 17 kV the particle size is about 2 μm . Reducing the current to 200 μA at 14 kV the particle diameter drops to less than 1 μm with a deposition rate somewhat less than 0.2 $\mu\text{m cm}^2/\text{min}$. At the lower currents, therefore, the possibility of using these sources for making complex patterns at submicron tolerances without masks looks extremely attractive. Since the material is deposited substantially as droplets, the writing speed would be markedly higher than that of systems using ions only. The crucial work will be on focusing and deflecting the spray.

S. ROBERTSON (University of California, Irvine) reported recent work at UCI on a novel mode of propagation of intense charge-neutral ion beams in magnetic fields. Two geometries have been considered. In one case the beam transverses a transverse magnetic field, analogous to that encountered by a beam incident in the outer magnetic surface of a tokamak. The motion is characterized by the value of $\epsilon = 1 - (\omega_{pi} / \Omega_i)^2$, where ω_{pi} is the ion plasma frequency and Ω_i the ion cyclotron frequency. In the second geometry, the beam is incident on a longitudinal magnetic field increasing with distance,

⁺ P. D. Prewett, L. Gowland, K. L. Aitken, and C. M. D. Manohy, *Thin Solid Films* 30, 117 (1981) - Appendix C.

corresponding to the situation in a magnetic mirror, for example. The important parameter governing the motion here is the ratio of the magnetic skin depth to the beam radius. Magnetic compression occurs if this ratio is much less than unity. For values much larger than unity, a short solenoidal field acts as a lens focusing the beam outside the field boundary.

Under conditions such that c/ω_{pe} is much less than the beam radius, the magnetic pressure gradient leads to beam compression until the transverse beam pressure balances the magnetic pressure. Experiments with a magnetic field of 700 G indicate compression leading to a current density increase from 1 to about 4 A/cm².

By changing the orientation of the magnetic field to longitudinal with a strength sufficient to make the electron (but not the ion) behavior radius smaller than the beam radius it becomes possible to focus a charge-neutral beam. It can be shown[†] that the focal length for both electrons and ions equals the geometric mean of the values for the ions and electrons separately. This is important in that it reduces the field required to focus protons, for instance, by two orders of magnitude.

The beam should not distort the imposed magnetic field. This leads to the inequality c/ω_{pe} much larger than the beam radius. For a nonrelativistic beam of radius 10 cm the current density at $v_0 = c/t$ is limited to 1 A/cm² in the lens.

Manipulation of the beam after neutralization may have advantages over the usual procedure in which the beam is focused, then neutralized, and allowed to reach the target ballistically.

[†] S. Robertson, H. Ishizuka, W. Peter, and N. Rostoker, University of California, Irvine, Physics Dept. Report No. 81-36 (June 1981) - Appendix D.

CONCLUSIONS

I. SOURCES CONSIDERED

A. GAS NOZZLE SOURCES (e.g., CAPILLARITRON)

Advantages

- . Large choice of gases
- . Simple construction
- . Quiet source
- . Current density up to 1 kA/cm^2 possible

Disadvantages

- . Large energy spread
- . Low gas efficiency

B. TAYLOR CONE LIQUID METAL ION SOURCE

Advantages

- . High Angular Intensity ($\sim 20 \text{ mA/sr}$)
- . Moderate energy spread ($\leq 20 \text{ eV}$)
- . Large ion choice
- . Small apparent source
- . Low noise at low currents

Disadvantages

- . Tip corrosion with some metals
- . Limited to wetting materials
- . Noise due to cone instability at high currents

C. CORNELL GAS PHASE FIELD ION SOURCE

Advantages

- . Very small virtual source (few \AA)
- . Small energy spread ($\sim 2 \text{ eV}$)
- . High angular intensity ($\sim 20 \text{ mA/sr}$)

C. CORNELL GAS PHASE FIELD ION SOURCE (cont'd)

Disadvantages

- . At present limited to H_2^+
- . Needs control of emitter surface configuration

D. THERMIONIC ION SOURCE

Advantages

- . Can produce negative ions
- . Small thermal spread
- . Large currents possible from extended source

Disadvantages

- . Limited ion choice
- . Source supply limited

E. SPUTTERING SOURCES FOR NEGATIVE IONS

Advantages

- . Small thermal spread (few eV)
- . Possibility of producing exotic ions
- . Good gas efficiency
- . Large currents
- . Many ions can be produced
- . Yield improvement with pulsed surface plasma source

Disadvantages

- . Formation process of negative ions not well understood

F. LIQUID METAL NOZZLE AND BLUNT WETTED NEEDLE SOURCES

Advantages

- . Large deposition rates
- . Controlled droplet size
- . Submicron droplet formation
- . High brightness
- . Compatible with ultrahigh vacuum applications
- . Available for gold alloys, silver, copper, aluminum alloys, and silicon
- . Highly directional

Disadvantages

- . Limited focusability of the charged droplets at present
- . Somewhat limited choice of materials
- . Particle size distribution possibly too broad for some applications

II. APPLICATIONS[†]

- . Direct (maskless) ion beam writing and milling (B,C,F)
- . Lithographic imaging at submicron resolution (B,C,F)
- . Image demagnification with extended source (B,C,F)
- . High resolution surface analysis (B,C,F)
- . Surface coating (A,B,C,E,F)
- . Irradiation of small targets by ballistic focusing (D,E)
- . Isotope separation within beams with small thermal spread (A?,B)

[†] The letters in parenthesis refer to the source classification of Part I.

The advantages of ion beams for many of these applications over alternatives such as electron beams and X-ray sources are well known. They are discussed in some of the references listed in Appendix A.

III. RESEARCH RECOMMENDATIONS

Present understanding about the operation of liquid metal sources is for the most part limited to qualitative conclusions drawn from highly idealized models. A great deal of numerical work needs to be done before quantitative predictions can be made.

Useful research directions include:

- . Investigate effects of needle geometry and surface microstructure on I/V characteristics, angular intensity, and energy spread.
- . Investigate energy spread as a function of emission angle; search for ways to reduce energy spread.
- . Study mechanisms of ion formation to distinguish between field ionization and field evaporation mechanisms; determine origin of neutral species for field ionization.
- . Investigate possible multiple Taylor cone array.
- . Develop new LMI sources for the elements, e.g., phosphorus and nickel.

The micro-droplet sources need further work on:

- . Understanding of relation between droplet size distribution and V-I.
- . Focusing and deflection schemes.
- . Calculation of cooling rates of very small objects.

The gas nozzle source requires:

- . Understanding of origin of energy spread.
- . Study of alternative geometries separating gas from ion beam.
- . Study of effect on ion production efficiency of various electron emitters coating the nozzle.

The Cornell gas phase field ion source requires:

- . Control of exact emitter configuration to obtain higher total currents.

Thermionics emitters require:

- . Understanding of surface treatment.

Sputter sources need:

- . Improved understanding of ion formation
- . Development of pulsed surface plasma source
- . Reduced energy spread.

APPENDIX A

A REPORT ON THE STATUS AND ACTIVITY IN RESEARCH AND APPLICATIONS
OF THE LIQUID METAL ION SOURCES

by

L. W. Swanson and J. Orloff

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Beaverton, Oregon 97006

In this report we wish to summarize the recent growth and current level of research and development relating to the liquid metal ion (LMI) source and applications. We are motivated to do this at this point in time in order that various groups in the USA will be aware what appears to be a unique source of a variety of ionic species that may have significant impact on microcircuit fabrication as well as other ion microprobe applications. The explosive growth of interest in the LMI source is becoming worldwide and is motivated by (1) a basic interest in the mechanism and characterization of the LMI source and (2) its potential for fine focus (i.e., submicron) applications.

From the existing work it can be concluded that a $\sim 0.1 \mu\text{m}$ beam of metallic (or non-metallic in the case of B and As) ions with a current density of 1 to 10 A/cm² is achievable. The use of low melting binary and ternary alloys extends the LMI source to a wide variety of ionic species. The ionic species which have been successfully employed thus far are summarized in Table I.

In Fig. 1 we summarize the growth of published papers on the LMI source and its applications. The very rapid growth observed in the last 2 or 3 years is due to a significant level of activity in this subject by Japanese and European research groups. Two of the authors

TABLE I

A Summary of the Ionic Species Employed in LMI Sources

| Ion | Pure Metal | Alloy |
|-----|------------|--------------|
| B | | x (Pt/B) |
| Li | x | |
| Al | x | |
| Ga | x | |
| In | x | |
| Cs | x | |
| Bi | x | |
| Sn | x | |
| Au | x | |
| Si | x | x (Si/Au) |
| Ge | | x (Ge/Au) |
| As | | x (As/Sn/Pb) |
| Pb | x | |

of this report, Drs. Ion Orloff and Lynwood Swanson recently visited the groups in Japan who are active in LMI R & D. Both of us were surprised at the extreme interest in the LMI source and the wide spread and, perhaps, coordinated R & D being devoted to this technology. The stated goal of the Japanese effort is to apply this technology to the development of microprobe systems for various aspects of microcircuit fabrication.

Similar activity pre-dating the start of the Japanese effort has been occurring in the USA and Europe, however, the level of effort in terms of manpower, funding and coordination has been exceeded by the Japanese program. We are not aware of such work in the USSR although our laboratory has been visited by Dr. E. I. Givargizov of the Institute of Crystallography, USSR Academy of Science who expressed considerable

interest in the LMI source work at OGC. A report of the Japanese trip by Dr. Jon Orloff is given in Appendix I. In Table II we summarize the various groups that are currently in LMI R & D and the approximate starting date of their activity.

Recently LMI sources have become commercially available. Dubilier Scientific, a French firm, has a license from Culham Research Laboratory to manufacture a Ga LMI source. More recently, Oxford Applied Research, a company founded by R. Clampitt, has announced a similar commercial LMI source of a variety of metals including Au.

The advances in applying the LMI source to microcircuit fabrication were first carried out at Hughes Research Laboratory (HRL) and to our knowledge they are still the leader in this technological application. However, based on the level of activity and commitment of the Japanese effort this technological advantage held by HRL may be overtaken. It should be kept in mind that many other applications of these sources in both fine focus and broad beam embodiment can be envisioned.

TABLE II

Summary of Groups Active in LMI Source Research and Applications

| <u>Country</u> | <u>Location</u> | <u>Principal Investigator</u> | <u>Approximate Start Date</u> | <u>Activity</u> | | |
|----------------|-----------------------------------------------|-------------------------------|-----------------------------------|-----------------|----------------|------------------------------------|
| | | | | <u>Source</u> | <u>Physics</u> | <u>Fine Focus Applications</u> |
| <u>USA</u> | | | | | | |
| | Phrasor Scientific Inc. Duarte, CA | J. Mahoney | 1974 | x | | |
| | Hughes Research Labs Malibu, CA | R. Seliger | 1976 | x | | x |
| | Oregon Graduate Center Beaverton, OR | L. Swanson, J. Orloff | 1975 | x | | x |
| | Pennsylvania State Univ. | T. Sakurai | 1978 | x | | |
| | Bell Telephone Labs Murray Hill, NJ | A. Wagner | 1979 | x | | x |
| | University of Chicago Chicago, Illinois | R. Levi-Strauss | 1980 | | | x |
| <u>U.K.</u> | | | | | | |
| | Culham Research Labs Oxford, England | P. Prewett | 1972 | x | | |
| | University of Cambridge Cambridge, England | A. Waugh | 1980 | x | | |
| | Oxford University Oxford, England | G. Mair | 1976 | | | |
| | Fullmer Research Institute | D. Stewart | 1976 | | | |
| <u>France</u> | | | | | | |
| | University of Paris, South | E. Collin | 1976 | x | | |
| <u>Japan</u> | | | | | | |
| | Osaka University | S. Namba | 1979 | x | | x |
| | Nippon Telephone & Telegraph | Y. Torii | 1980 | | | x |
| | Electrotechnical Laboratory | M. Komuro | 1979 | x | | x |
| | Hitachi Central Res. Labs JEOL | T. Ishitani S. Ohta | 1979 | x | x | x |

APPENDIX I

Trip Report

Visit to Japan

by

Dr. Jon Orloff

November 1980

Report on Visit to Japanese Laboratories, November, 1980

Introduction

In November, 1980 I visited several laboratories in Japan where research on electron optics and liquid metal field ionization (LMI) sources is under way. The laboratories were: Osaka University; Nippon Telephone and Telegraph (NT & T); The Electrotechnical Laboratory at Tsukuba (Science City), Ibaraki; Japan Electron Optics Laboratory (JEOL); Hitachi Central Research Laboratories (CRL). At each of these laboratories, with the exception of JEOL, there is intense interest in and research devoted to the application of LMI, particularly towards microfabrication. At JEOL the interest is on applications with a view towards producing ion-optical instruments. A brief description of what I was shown follows.

Osaka University

Professor Namba heads a group studying LMI sources. They are working intensively on As sources and have two which provide As beams: B-Ge-Pt-As and As-Sn-Pb. They are also working on an As-Se eutectic with a 180°C melting point. This group has some six or eight graduate students who are making sources and measuring their properties.

Professor Goma, who works closely with Professor Namba, is designing and building an optical column to utilize LMI sources. It consists of two einzel lenses, a Wien filter for mass separation and an octupole stigmator deflector. This laboratory had just received a \$150,000 grant from the Japanese government for focussed ion beam studies.

There are extensive facilities at Osaka University for the study of microcircuit fabrication, including MBE equipment, a JEOL Model 50 e-beam machine, sputter-etch capability for resist development,

light-optical lithographic devices and wet chemical development facilities.

It is evident that the University is training large numbers of students, at the graduate level, in the techniques of microcircuit fabrication as well as in electron optics and field ionization techniques.

I was surprised at how crude the equipment and facilities were at the University in comparison with the private company laboratories. Much of the equipment used by graduate students and professors alike looked like what one would expect to find in freshman laboratories in the U.S.

NT & T

NT & T is the Japanese analogue of Bell Labs. They have a great interest in microcircuit fabrication and have elaborate facilities for experimental work on their production. They have just begun to study focussed ion beams produced with LMI sources, using a Dubilier source (including power supplies). This is coupled to an optical column which consists of an einzel lens followed by an immersion lens, a beam blanker and a deflection system. They have achieved 1 μ m beam size with currents of 0.7 to 0.8 nA in preliminary tests.

The NT & T group is studying means of developing resist exposed by ions. They came up with the O_2 plasma technique in which a layer of Ga_2O_3 , formed from Ga implanted in the resist, protects the underlying resist from attack by the plasma. In effect, the implantation of Ga creates a negative resist (H. Kuwano et al., Jap. J. Appl. Phys. 19(1980) 615). The ion shower concept, in which a collimated beam of

halocarbon ions produced by an rf field is accelerated to 1 kV and aimed at the surface of the resist, is being tested extensively. It is expected that techniques such as this will be used with ion beam resist exposure systems. Workers at NT & T commented that they believed ion resist exposure systems held great promise for construction of submicron features.

Electrotechnical Center, Tskuba, Ibaraki

Tskuba, or science city, is the location of several large, new laboratories. There are several thousand scientists and technicians who live near and work in this government supported facility. I visited only the section involved with LMI studies. It took about six weeks to obtain permission to see the laboratory.

The group studying electron optics and ion beams at the Electrotechnical Center consists of some half-dozen professional level individuals. They are young and aggressive and are vigorously studying new electron optical schemas. In particular, they are examining those which involve the use of electrostatic quadrupole lenses with entrance and exit apertures excited to potentials chosen to minimize spherical aberration. Application of strong focussing lenses to ion beams is foreseen. Work on electron optics is both theoretical and experimental, as is work on LMI sources. Efforts are now being made to study the exposure of resist by ion beams using conventional ion sources. A 50 kV system with an LMI source, beam blanking capability and current stabilization by means of a control electrode in the ion gun, is in the design stage.

The stated goal of this group is to apply fine-focussed ion beams to microcircuit fabrication. There are extensive facilities available to them for I.C. fabrication by conventional means, which they hope to augment by this new technology. Any successful developments can be expected to be rapidly utilized by Japanese industry, since this is a government funded facility.

JEOL

JEOL is primarily a manufacturing and engineering company which does relatively little research. JEOL produces a broad line of electron microscopes and other scientific equipment, including a scanning Auger microscope and an electron beam lithography system. Although it is not a research-strong company, it has demonstrated great skill at exploiting research developments made elsewhere. At present, a low level effort is under way to study LMI sources. If there should develop a market for ion beam instrumentation based on reasonably sophisticated optics, it would not be surprising if JEOL were to build and sell such devices, applying the knowledge it gained from its e-beam machine program.

Hitachi

Hitachi is a large (1979 sales $\approx \$7.7 \times 10^9$) company which spends about 5% of its sales on R & D. I visited both the Central Research Laboratories (CRL) in Tokyo and the Production Engineering Laboratories in Yokohama. The latter organization is interested in the use of fine-focussed ion beams for microcircuit fabrication, but has done no work on the problem. The CRL groups are actively engaged in research on sources and optics. Two types of e-beam lithography machines have been developed at CRL, for internal Hitachi use. One

is a field emission, round beam, vector scan system and the other is a LaB₆ cathode, shaped beam system along the lines of those developed at IBM by Pfeiffer and coworkers. Therefore, the expertise needed to build sophisticated electron-optical columns clearly exists at CRL. Although I was invited to visit CRL and I spent time seeing the e-beam systems, I was shown very little of the experimental systems used for LMI studies. Whether this is because they are very secretive or because they have done relatively little, I don't know. Theoretical studies are under way on the nature of the source, and there was certainly great interest evidenced in the use of ion beams for resist exposure. Questions asked of me relating to practical applications of the LMI source indicated that an experimental program was just beginning. Hitachi personnel indicated that they were committed to pursuing all approaches to submicron lithography. The size of the effort (I estimate 5 Ph.D. level people) and their high quality personnel surely mean that, if it is possible to apply LMI techniques to microcircuit fabrication, Hitachi will do it.

APPENDIX II

BIBLIOGRAPHY OF LMI SOURCE ARTICLES

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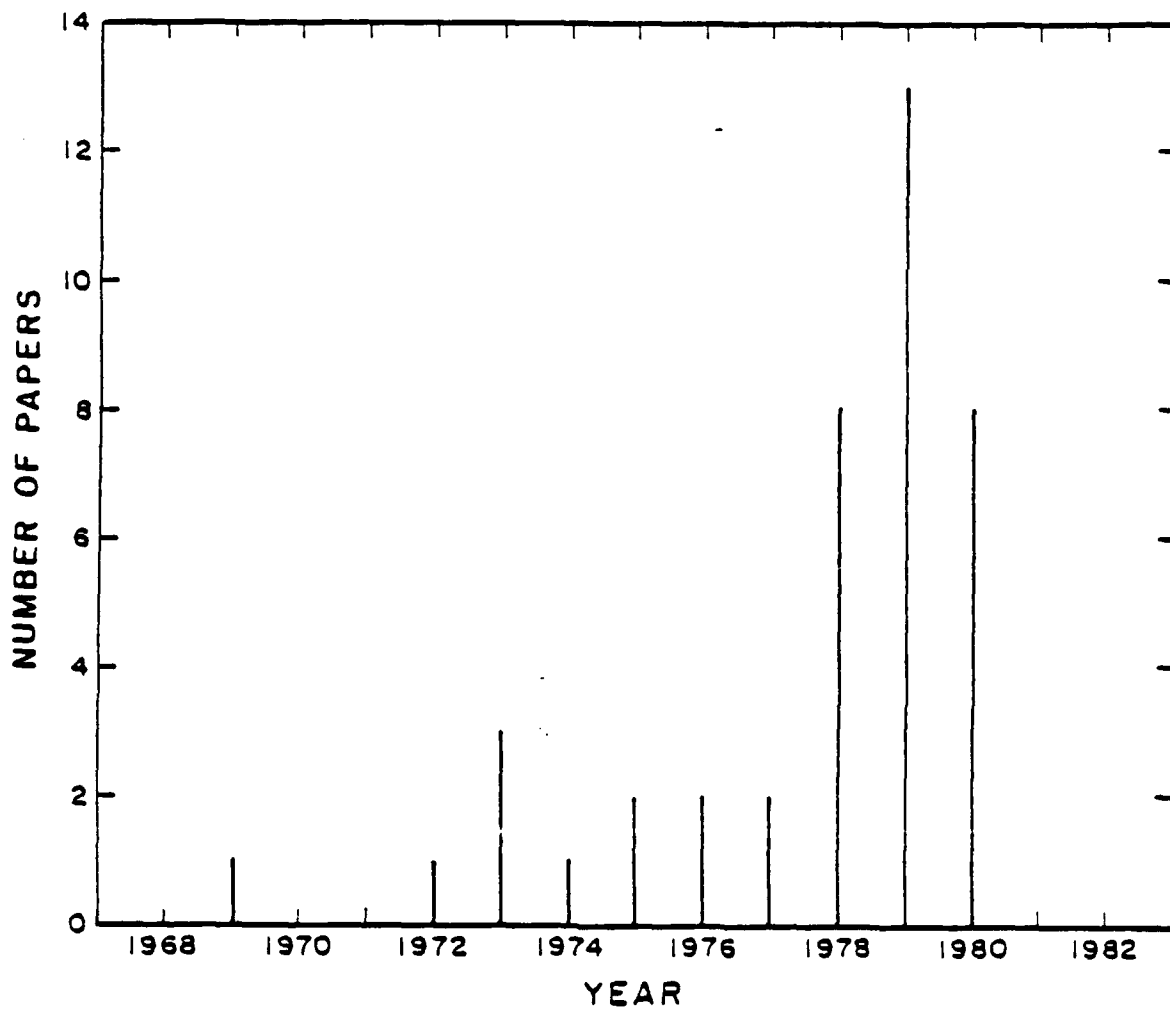


Figure 1. Number of published papers on LMI source and applications vs. time.

APPENDIX 8

ENERGY SPREADING IN THE HYDROGEN FIELD IONIZATION SOURCE

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Abstract

The application of field ionization as a tool for routine submicron fabrication will depend critically upon the characteristics of the source; the angular intensity and the energy spread of the ions. The chromatic aberration coefficient of the ion optical system, the angular aperture and the energy spread combine to set the limit of spot size and current density for very high resolution probes with sources of energy widths even as low as 1 eV. It is known that the various molecular species produced by hydrogen field ionization are identifiable by the energy distribution and because the mass of the ion will determine the particle range in resists at any given energy, energy measurements carry further importance in the development of the probe forming system. In order to characterize the source energy widths and mass species as a function of beam current, angular intensity and surface characteristics of the emitter tip, we have designed and operated a unitized, positionable intermediate image filter lens with a demonstrated resolution of 0.1 eV at 3.5 KV and acceptance half-angles of less than 9 mr. At angular intensities less than 10 $\mu\text{a}/\text{sr}$, 90% of the field ions from bright site emission of fields of $1.5 \text{ v}/\text{\AA}$ occur within an energy width of from 1.2 to 1.9 eV. This narrow spread shows that only H_2^+ is being produced. The beam current and angular intensity, from a single bright site can be increased by raising the source gas pressure and the applied field (-6%). That energy broadening occurs at high intensities is demonstrated by the fact that at 3.5 $\mu\text{a}/\text{sr}$ (0.5 na emitted through 50 μm aperture subtending a half-angle ($\theta_{1/2}$) of 0.0067r) 90% of ions appear within 1.9 eV (0.94 eV FWHM) while at

18 $\mu\text{a/sr}$ (2.5 na, $\theta_{1/2}$ of 0.0067r) 90% of ions appears within 2.2 eV (0.98 eV FWHM) where a tail develops on the low energy side causing a broadening of the energy distribution.

The impact of field emitter surface configuration on source angular intensity and reliability are also discussed.

Small, intense ion sources have important potential applications in microfabrication and microanalysis. The utility of the ion microprobe for submicron work is presently limited by source brightness and secondary ion collection efficiency. Ion microprobe systems designed for x-ray fluorescence analysis are of interest because of the higher resolution (region from which the x-rays are emitted would be determined by beam size below 0.1 μ m dimensions) and higher signal to noise (no primary beam bremsstrahlung) of ions as compared with electron probes. With a high brightness, liquid gallium source, Seliger, et al,⁽¹⁾ have demonstrated the potential of ion probes for use in microfabrication. This demonstration utilized the inherent high sensitivity and high resolution of ion resist exposure and ion beam machining.

The development of high brightness ion sources with low energy spreads are of interest because chromatic aberration limits beam size and deflection fields at high resolution (probe diameters of 0.01 - 1 μ m). Parent ions from field ionization have been observed to have energy spreads of ~ 1 eV at threshold fields.^(2,3) Ion beams of such ions would have potential advantages when compared with liquid metal ion sources which have minimum energy spreads of 4.5 eV.⁽⁴⁾ This fact and/or the inherent high brightness of the field emitter configuration has stimulated interest^(5,6,7,8) in the process of field ionization as a potential source for high resolution probe forming systems.

In addition to source brightness and energy spread, mass spectral purity is an important source characteristic because ion range depends strongly upon the mass of the probe particle. It is known that field ionization at fields of 1 - 1.5 v/ \AA produces parent ions, H_2^+ , and as the field is increased to 2 v/ \AA and above, a mixture of H_2^+ parent ions, H^+ secondary ions and H_3^+ with energy spreads which increase rapidly from 4.5 eV with increasing fields.

The final point of inquiry in the gas phase source investigations is that of whether the process can be controlled to be sufficiently flexible to be usable over a large range of probe system operating parameters (sizes 0.01 to 1um) and reliable and stable for extended periods. To achieve the later characteristics, control of the emitter surface configuration is the important consideration.

Ion Source Operation.

Because the field emitter configuration is one which produces a virtual point source of ions, the source brightness may be inferred using measurements of the ion angular current intensity obtaining the emission area either from ion micrographs or calculated estimates of the field-emitter cross-over radius. The angular current intensity is measured in these experiments using a 50um defining aperture in the energy analyzer to define the angular divergence of the beam, measuring the transmitted current on the image intensifier with the analyzer grounded. The size of the projected image a known distance from the emitter gives the subtended angular aperture (see Figure 1). There is an auxiliary electrode on the intensifier on which a negative 180V is applied for the purpose of suppressing secondary electrons from the intensifier surface which would otherwise double the apparent ion current. In these measurements of the source angular intensity, no correction is made for the lens effect of the cathode aperture which decreases the apparent angular intensity because of the axial field difference across the defining aperture.

An understanding of the mechanism of high brightness emission in the gas phase source is acquired using the analysis of Southon⁽⁹⁾ for the supply function of neutral molecules to the high field region for a hypercolored emitter expressed by the formula:

$$S = \frac{AP}{2k} \sqrt{\frac{E}{V}} \quad \text{(particles/sec)}$$

where F is the electrostatic field, T is the temperature of the gas and the emitter, A is the area of the emitter surface in the high field region (a hemispherical cap of radius R) $= 2\pi R^2$, P is the source gas pressure, k is the Boltzmann constant, α is the gas polarizability and M is the atomic mass of the gas molecule. Although the formula underestimates the current we observe from the emitter, it qualitatively describes the impact of the physical parameters on the supply of neutrals and therefore the current. It is clear the source should be designed for low temperature with as high a gas pressure as possible without inducing ion-neutral beam broadening. Also the emitter surface should be configured to maximize the area of the field region.

Conversion of the neutral supply to localized, high brightness emission occurs by diffusion in the high field region to locations of higher field where ionization times are short. Figure 2 shows an example of this effect using a tungsten $\langle 110 \rangle$ emitter which has been flashed at $\sim 2000^\circ\text{K}$ with some residual partial pressure of hydrogen present. In figure 2a, the ion pattern from the emitter apex region is viewed through an aperture subtending $\sim 0.5^\circ$. Figure 2b shows the emission pattern from the same emitter (pressure, temperature, intensifier gain and emitter voltage held constant) after the appearance of a bright site interpretable in this case to be a protruding contamination (N_2 , O_2 , etc.)-tungsten complex on the surface.⁽¹⁰⁾ It is observed that not only is the total current through the aperture increased because of neutral supply from regions subtended by the aperture, but the original emission pattern is of lower intensity particularly near the bright site. Clearly, there is diffusion of the neutral supply in the high field region of the emitter which can be interpreted as occurring in a physisorbed layer not totally equilibrated to the emitter temperature because of the large cur-

rents emitted and the relatively large polarization energy being dissipated (0.07 eV per particle). The practically complete localization of emission from smooth, diffuse ion patterns of large angular apertures with the appearance of a contamination site or a negative ion sputter damage site on the surface of emitters with radii of 600-900 Å indicates that material is diffusing distances greater than 400 Å.

These particular emission sites, produced by contamination or negative ion bombardment, are of limited utility because there is no control over their location on the emitter surface nor on their formation. Over several hours of source operation other bright sites will appear at a rate determined by the ion current and contamination partial pressures. These competing sites have the effect of reducing the supply to the original site and therefore, the current at the ion beam emission site being used as a source of H_2^+ ions. The configuration reported by Brady, et al.⁽¹¹⁾ minimizes this effect by producing a localized high field region on the axis of <100> oriented single crystal tungsten emitter and therefore, long range effects of surface rearrangement are not as pronounced and local rearrangement is not as likely compared to the annealed emitter bright sites. Although high source pressures are required, at the present time this emitter is the most appropriate source configuration for obtaining a steady H_2^+ ion source with angular current intensities of up to ≈ 15 μ A/sr.

Surface features from 1 to 10 atoms in size, see Figure 2b, such as expected from field induced tungsten-nitrogen complexes or random clusters are capable of sustaining maximum current densities corresponding to 20 μ A/sr before they are excited and desorbed, or restructured. Therefore, reliable operation is limited to ≈ 15 μ A/sr both in this case and also in the case of the pointed emitter described by Brady, et al.⁽¹¹⁾ which is supply limited.

Our investigations into the possibility of reliably generating structures of larger molecular complexes, or protruding clusters, on annealed field emitters have on occasion demonstrated higher angular intensities in which the structure is not desorbed with higher total currents. However, after investigating the possibilities of microcrystal growth, metal whisker formation, radiation damage, thermal dissolution of net planes and thermal field effects⁽¹²⁾ we have found these processes to be unreliable methods for producing a surface configuration which localizes the emission on axis without subverting the neutral supply mechanism. We are presently investigating the possibility of fabricating a low aspect ratio structure $\sim 20 \text{ \AA}$ high and 400 \AA wide on a field emitter 5000 \AA in radius. Techniques to be investigated for providing the local field enhancing emission surface are SEM contamination cones, metal layers produced by microfabrication techniques⁽¹³⁾ using resists such as NaCl , MgF_2 , or contamination cones as resists for etching (electropolish or plasma) or deposition (electroplate), SEM radiation deposited tungsten from a layer of adsorbed $\text{K}(\text{CO})_6$, etc. Such a protruding structure would contain 10^5 atoms and 10^4 surface atoms on which surface roughening due to field-enhanced contamination complexes which occur at a rate of several per hour would be insignificant. With good adhesion or structuring in tungsten, desorption of the surface material does not occur. Total currents increase in proportion to the ~ 2.5 power of the radius (see Eq. 1) and reliable operation at 60 \mu A/sr can be achieved.⁽⁸⁾ A fabrication technique also offers the possibility of controlling the ion emission angular aperture.

Ion Energy Analysis.

For the purpose of having an energy analyzer of simple design which would be removable for ion pattern viewing and positionable for alignment on any

particular emission site, an intermediate image filter lens (retarding analyzer) was designed using the analysis and data of Simpson and Soa^(13,14) to have ~ 0.1 eV resolution at 5KV and 10mr acceptance half-angle. Figure 1 shows the electrode configuration and aperture sizes. The aperture spacings are 4.4mm and 0.9mm. The analyzer was designed for an image in the retarding plane with an object distance of ~ 3 mm in front of the cathode aperture when the emitter, bias electrode and retarding plane are at essentially the same potential.

There is generally the perception that space charge effects will dominate ion beam optics particularly in the source and in a retarding energy analyzer which has a low energy cross-over. Knauer⁽¹⁵⁾ has analyzed energy broadening in ion beams which is due to the "thermal" Boersch effect and Boersch effect which is the result converting transverse velocity of the cross-over motion into longitudinal energy.

The "thermal" Boersch effect will not be considered here because the transverse beam temperature is extremely small and not well defined. The initial kinetic energy of ions produced by field ionization is very low as a result of the neutral supply accommodation process. The thermal kinetic energy at 10^4 K, kT , is ~ 1 meV. Because the neutral supply is not in equilibrium this low value is not reached, however, ~ 10 meV is a reasonable, conservative estimate for the average transverse initial energy of the resulting ions. This small value is further reduced by the acceleration of the particles out of the high field region which has central field properties.

Two further regions of beam energy broadening which are analyzable in terms of the formulation of Knauer⁽¹⁵⁾ are those of the diverging region in front of the source and the converging region in front of the retarding

plane of the energy analyzer. The energy analyzer region is the most critical because of the relatively larger region of low ion beam energy there as compared with the corresponding region in front of the field emitter where the field of 1.5 v/\AA rapidly accelerates the ions.

Figure 1b shows the trajectory boundaries in the lens. In operation the analyzer would produce the lowest energy spreading if the lens imaged the source behind the retarding plane. In such a case, the limitation in energy resolution due to variation of the saddle potential between the axis and a radial point at the edge of the beam boundary would occur if the cross-over were too far behind the retarding plane. The precise operating point was found by adjusting the bias high and low observing the values at which diminution of energy resolution occurred. A -1% bias was found to give the highest resolution. It should be noticed that the acceleration lens has no effect upon the energy analysis because it serves only to reaccelerate the transmitted particles to the image intensifier-detector.

Using the expression of Knauer⁽¹⁵⁾ for the root-mean-square energy spread for the upstream beam boundary of the lens cross-over, energy spread resulting from the lens operating with 2.5 na into 6.7 mr (Kinetic Energy - 0.1 eV) is 0.01 eV. This rather small value results from several effects: (i) The half-angle of the cross-over is very large, 0.28r for a 6.7 mr entrance angle as calculated using the Helmholtz-Lagrange relation. The root-mean-square energy spread is inversely proportional to this angle. (ii) The area of the beam in the retarding plane can be relatively large. A calculation of the distance from an axis point to an off-axis point in the aperture of the lens for which the potential varies by 0.1 eV as a result of field penetration shows a beam diameter of 3 μm . (iii) Beam interactions have root-mean-square energy spreads

which depend upon the fourth root of the particle mass. Thus it is clear that for the investigation of source properties, the intermediate image filter lens configuration provides both positionability and energy resolution.

Using field emitted electrons from a thermally annealed $\langle 110 \rangle$ field emitter as a calibration standard, the energy resolution of the analyzer can be estimated. Figure 3a,b show electron current for 2 volt scans of the retarding plane and bias voltages in the form of photographs of the oscilloscope display of the electrometer current from the intensifier as a function of applied scan voltage. The data shown in Figures 3 and 4 are unprocessed. Integral distributions of current transmitted through the lens as the retarding plane voltage is manually scanned once during a time period of ~ 1 min. The apparent resolution of 0.1 eV shown in Figures 3a,b at an electron energy of 3.5 KV gives a demonstrated lens resolving power of 2.8×10^{-5} . At 7 KV, the ion energy resolution is at least 0.2 eV which is adequate for measuring ion energy widths of 1 eV and above.

Figures 3c,d show the effect of localized high brightness emission and increased pressure for such emission on the energy distribution of the ions. These data show the expected energy distribution of parent H_2^+ ions produced by field ionization. At $3.5 \mu A/sr$ ($0.5 nA$, $\theta_{1/2} = 6.7 mrad$) and $18 \mu A/sr$ ($2.5 nA$, $\theta_{1/2} = 6.7 mrad$), peaks of essentially 1 eV (FWHM) are observed. At higher angular intensities, Figure 3c shows the presence of energy spreading in the form of a low energy tail which is accounted for by several physical processes. (i) Pressure broadening between the emitter and the analyzer defining aperture. At chamber pressures of 5×10^{-6} and above, source pressure and collision cross sections indicate the probability of ion-neutral collisions (sum of H_2 and above source pressure). (ii) Charge interaction of ions and ion-neutral

interaction in the region of the ionization zone near the emitter surface. Although, as mentioned above, the field is quite high (1.5 v/\AA), it is seen from the analysis⁽¹⁵⁾ that the mean-square energy spreading is proportional to the beam diameter and the current density which is proportional to the emitted current divided by the diameter of the beam. Since current densities are high, or beam diameter is extremely small, for a short distance near the ionization zone source energy spreading may result in addition to some collisional broadening resulting from high neutral density in the same region.

Figure 4 demonstrates the importance of proper adjustment of the source electrostatic field. There is energy broadening which is apparently due to chemical reactivity at bright sites operated at fields below those sufficient to produce a large amount of field dissociation. This result is identified as a surface chemical process because of its pressure dependence, i.e., the amount of products H^+ and H_3^+ , depends upon the number of neutral hydrogen molecules present at the ionization site. Because of its detrimental effects upon the source optical properties, this regime is to be avoided in source operation by operating the source on the low field side of the surface supplied peak in the total ion current. In this field window, only H_2^+ is produced as is seen in Figure 3.

Gas phase field ionization at $18 \mu\text{a/sr}$ produces ions with an energy spread of 1 eV (FWHM) if the field is adjusted properly. The brightness of the source can be estimated using either the emission area from the field observed in the field ion pattern or from estimates of the cross-over radius. A conservative estimate of the initial, transverse ion energy of 0.05 eV predicts a cross-over radius (formulation of Wiesner and Evernart⁽¹⁶⁾) which is 5-7 \AA for the tip radii (600-900 \AA) used in this work. Source brightness of $2 \times 10^3 \text{ a./cm}^2 \text{sr}$

at 5 KV are calculated. With a gun lens characterized by chromatic and spherical aberration coefficients $C_s = C_c = 2$ cm, the apparent source probe formed would be chromatic aberration limited using an acceptance half-angle of 5 mr, giving a probe radius of ~ 100 Å with a current density of 700 a/cm². The calculated effective brightness is 6×10^7 a/cm²/sr at 50 kV assuming a source of 18 µa/sr angular current intensity and an energy spread of 1 eV FWHM. These results compare well with the liquid metal sources which operate with 4.5 eV energy spreads at 20 µa/sr which give an effective brightness with the assumed lens of 4×10^6 amp/cm²/sr at 50 kV using a 5 mr acceptance half-angle and plasma sources which have source brightness of only $\sim 10^3$ a/cm²/sr with energy spreads of at least 5 eV.

Acknowledgment.

This work has been supported by NSF Grant 77-09688 through the National Research and Resource Facility for Submicron Structures at Cornell University.

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Figure Captions

Figure 1. a) Schematic diagram of the source showing field emitter and cathode aperture which are cooled to temperatures of 10-15°K. The intermediate image filter lens shown gives integral energy analysis of the ion beam. It is mounted so as to be completely positionable and removable for ion pattern viewing. Beam current is measured and observed on an electron intensifier (CEMA) which has an electron suppressor ring for eliminating secondary electron currents from the ion current measurements. b) Ray path of ions during energy analysis. Ions emitted into an acceptance half-angle of 6.7 mrad cross the retarding plane at an angle of 0.28 rad.

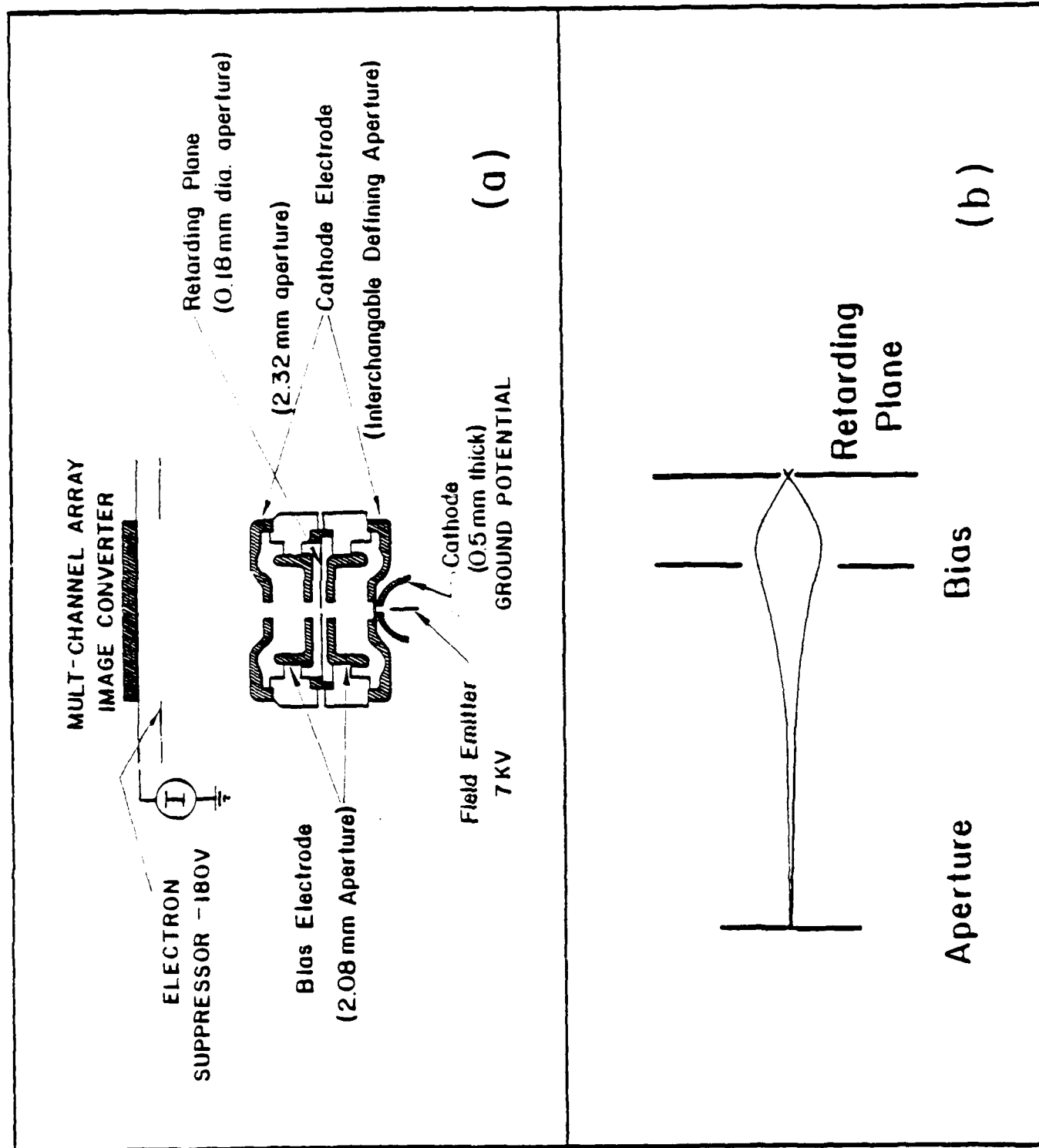
Figure 2. Ion emission from a localized bright site on a thermally annealed tungsten <110> oriented field emitter. a) Ion pattern from smooth surface (7KV, 7×10^{-7} torr chamber pressure, 6.6 na through cathode aperture subtending 0.5 rad). b) Bright site emission (7KV, 7×10^{-7} torr, same intensifier setting as 2a), 13 na through cathode aperture). Angular current per unit source pressure depends upon the number of other emitting sites and the shape of the emitter endform. Current appears localized into half-angles of ~10 mrad at a maximum of 20 na/sr.

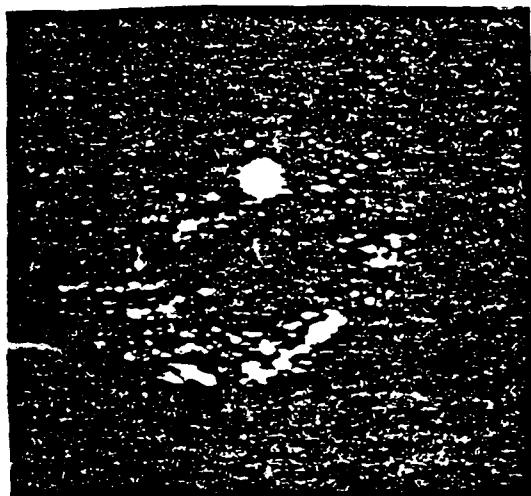
Figure 3. a) b) Field emission electron energy distribution (integral) calibration of the energy analyzer. Electron current of 6×10^{-11} a is collected through 50µm aperture subtending ~9 mrad half-angle. Energy widths of electron emission are 0.2 to 0.3 eV which is observed using the analyzer demonstrating a resolving power $\Delta V/V = 0.1 \text{ kV}/3.5 \text{ kV} = 2.2 \times 10^{-5}$. c) Ion energy distribution (integral) at 18 na/sr (2.5 na, $\theta_{1/2} = 6.7 \text{ mrad}$), 0.98 eV FWHM, 2.2 eV for 90%

of ions. d) Ion energy distribution at 3.5 $\mu\text{a/sr}$ (0.5 na , $\theta_{1/2} = 6.7 \text{ mr}$), 0.94 eV. The increase in current of c) is the result of increasing the source pressure and increasing the voltage ~5% from that of d). Ion emission from these sites is limited by desorption to a maximum of 20 $\mu\text{a/sr}$. Ion emission from the pointed emitter configuration discussed in the text is limited to 15-20 $\mu\text{a/sr}$ by breakdown at the high source pressures required.

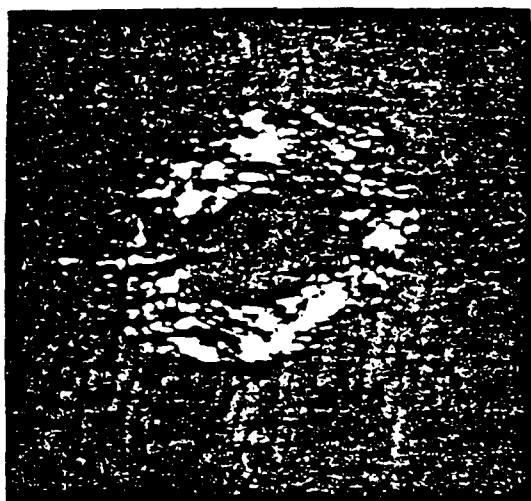
Figure 4. Pressure effects in the energy distribution of bright sites operated at too high an average electrostatic field ($\sim 2 \text{ v/\AA}$).

- a) Chamber pressure 5.5×10^{-7} torr.
- b) 3×10^{-7} torr
- c) 9×10^{-8} torr

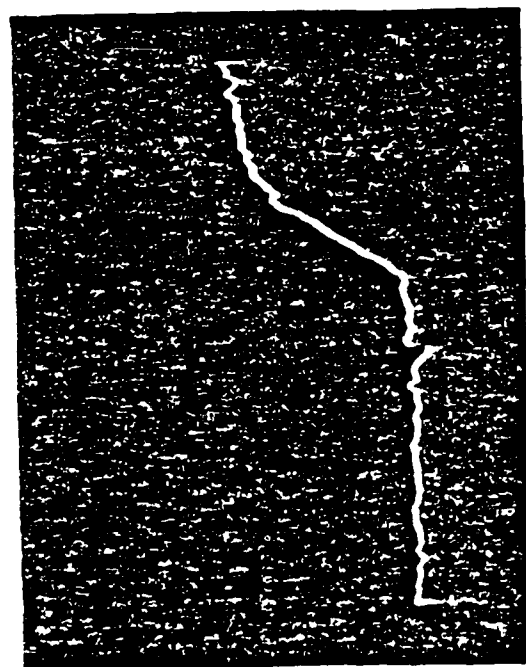




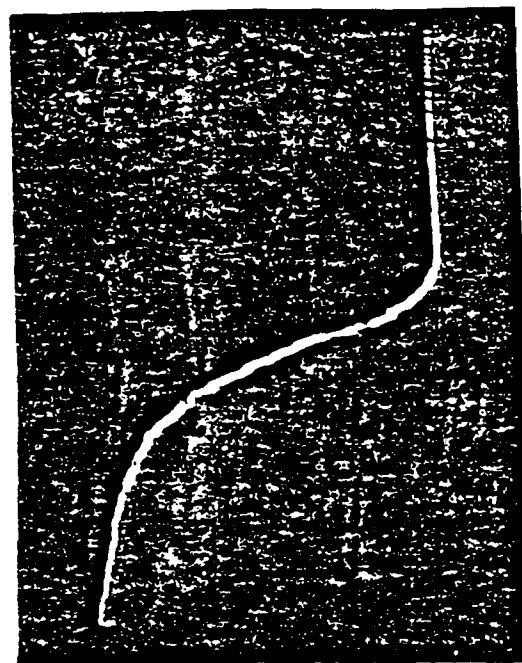
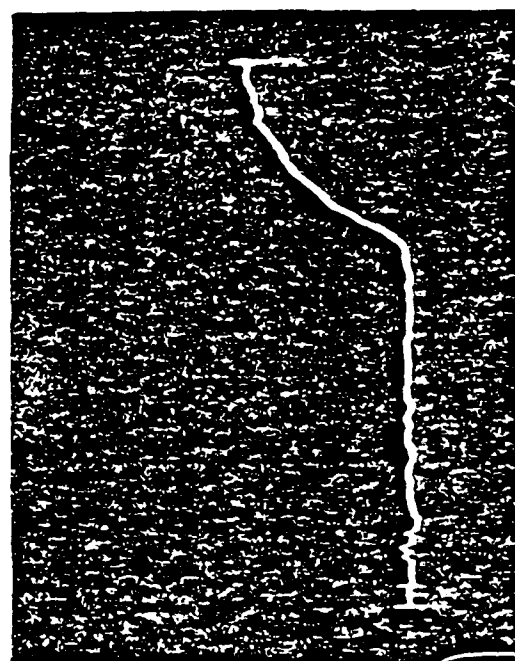
b



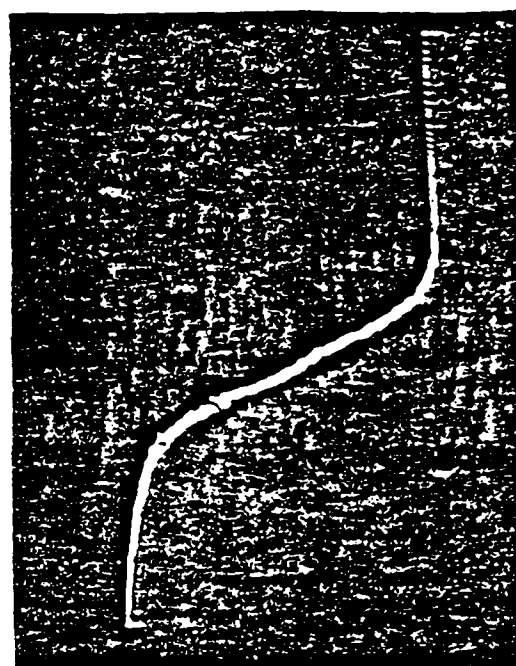
a



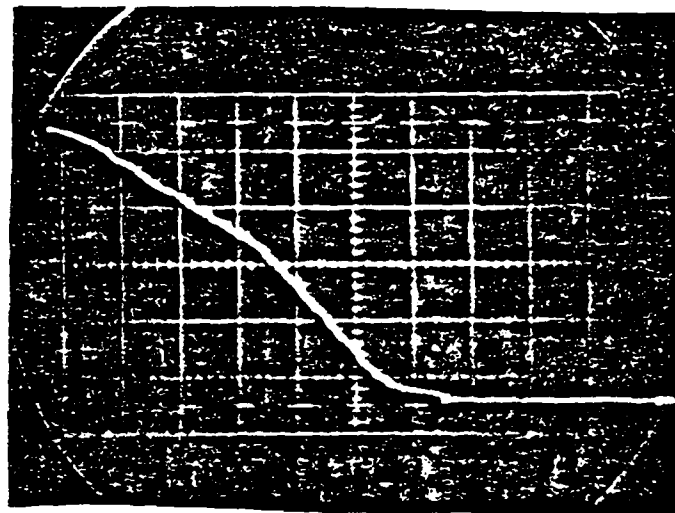
0.5 0 -0.5
ENERGY (eV)



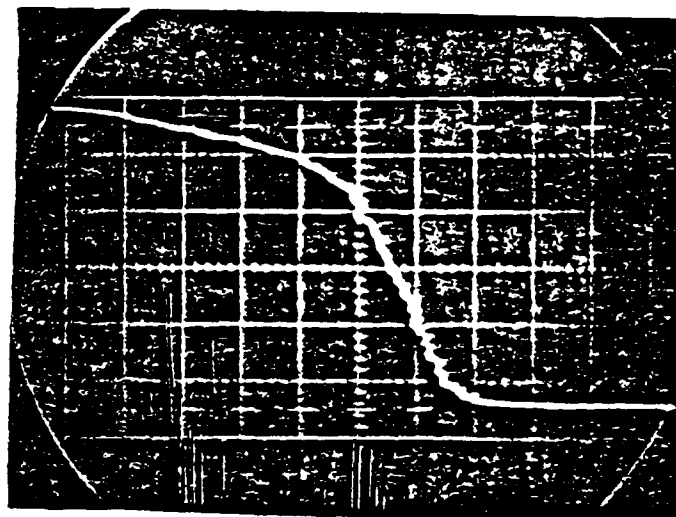
-5 0
ENERGY (eV)



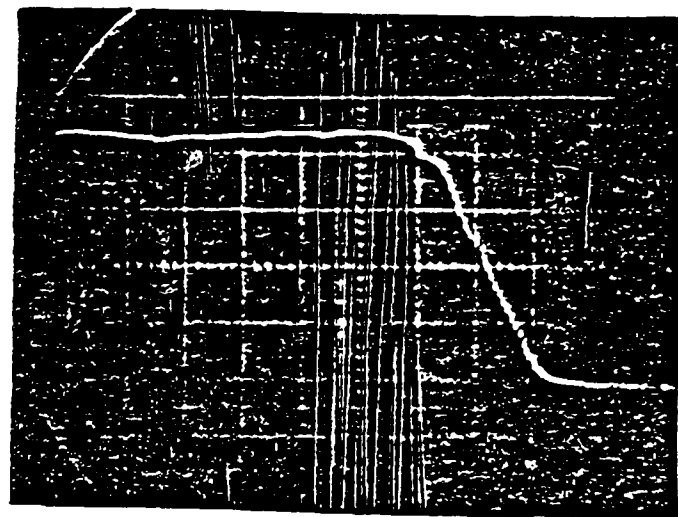
a



b



c



-5 0

Energy (eV)

APPENDIX C

THE DEVELOPMENT OF A SPRAYER FOR FIELD EMISSION DEPOSITION*

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In the process of field emission deposition (FED) or IONCOTE, a blunt wetted needle is used to generate a coating spray of ions and droplets under the action of a high electric field. Coatings of a variety of elements and alloys can be produced in this way, but this paper deals mainly with gold for which most progress has been made.

The blunt needle concept was incorporated into the design of a prototype sprayer and its performance characteristics were investigated. In particular, control of emitted droplet size was achieved and the optimum operating conditions with a droplet diameter of about $2\ \mu\text{m}$ were identified. Under these conditions, coating rates in the range $0.5\text{--}1.0\ \mu\text{m cm}^2\ \text{min}^{-1}$ are achieved with measured adherence as high as $2 \times 10^7\ \text{N m}^{-2}$ for coatings of gold on glass.

Preliminary experiments demonstrated electrostatic focusing and deflection of the coating spray and spot sizes of a few millimetres in diameter were achieved. As a result the FED process shows great potential for maskless generation of coating patterns such as gold-on-alumina interconnects for hybrid microcircuits.

1. INTRODUCTION

There is currently a great deal of interest in the development of high brightness liquid metal ion sources for a variety of applications such as the lithography of microcircuit patterns and high resolution surface analysis¹. One such source (DSL Series A and B ion sources, Dubilier Scientific Ltd., Abingdon) relies upon the emission of ions from an electric-field-distorted film of liquid metal which is anchored by wetting to the surface of a needle of tip radius $r_t \approx 5\text{--}10\ \mu\text{m}$. In this paper we describe the development of a closely related "field emission system" in which a greater supply of liquid metal is employed to provide a beam composed of a mixture of metal ions and charged liquid droplets. This spray of ions and droplets may be used to produce highly adherent coatings of a variety of metals and alloys as has been reported²⁻⁴.

* Paper presented at the 3rd International Conference on Ion and Plasma Assisted Techniques, Amsterdam, The Netherlands, June 30-July 2, 1981.

This field emission deposition (FED) or IONCOTE process (IONCOTE is the registered trademark of Dubilier Scientific Ltd., Abingdon) shows potential for a variety of applications in which localized highly adherent coatings of high structural integrity are required. Applications investigated to date include electrical contact patterns, corrosion protection and metal bonding. In contrast with conventional ion plating, the FED technique is a true vacuum ion process compatible with ultrahigh vacuum conditions. The coating flux is highly directional and uniform deposits are produced by mechanical manipulation of the substrate. In previous experiments patterned coatings have been obtained by the use of masks, but recently we have been able to demonstrate that both ions and droplets can be focused and steered by electrical means. As a result the FED or IONCOTE technique now appears to offer considerable potential for maskless generation of complex coating patterns. This will be discussed more fully in Section 3.

2. SPRAYER DESIGN

We shall refer mainly to the use of gold as the coating material since this is the element on which most effort has been concentrated. Recent progress has been made, however, using sprayers of gold alloys, silver, copper, aluminium alloys and, in particular, silicon (see Pang *et al.*⁵).

The basic design requirements are that a reservoir of liquid metal should be maintained at its m.p. and subjected to a high electric field by application of a positive d.c. potential between the reservoir and a negative counterelectrode or extractor as shown schematically in Fig. 1. The required temperature (1060 °C for gold) is achieved by means of a resistively heated furnace and the reservoir may take a variety of forms including an open nozzle or capillary (Fig. 1(a)). In practice, we have found that the wetted needle design, similar to that used in liquid metal ion sources⁴ and shown in Fig. 1(b), produces the most controllable performance characteristics together with coatings of an acceptable quality (see Section 3). It is essential that the liquid should wet the surface of the needle to form a film which is anchored to it and which is maintained at the needle tip by flow over its surface from the reservoir. It is vital that this flow should be controlled in order to prevent flooding of the tip. As we shall see in the next section, an excessive supply of liquid to

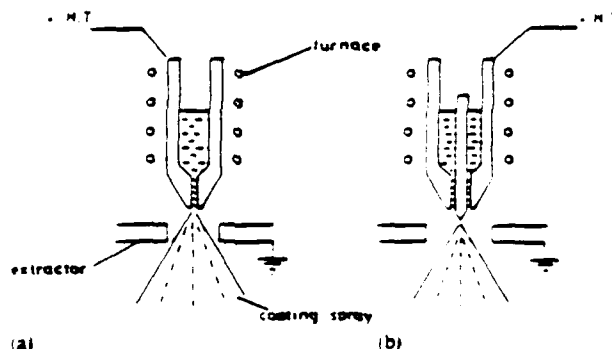


Fig. 1. Schematic diagram showing the basic sprayer design using (a) open nozzle or capillary and (b) wetted needle configurations.

the tip causes the emission of droplets which are too large ($d > 2 \mu\text{m}$) to produce high quality coatings. An inadequate flow, however, will starve the tip, thereby reducing the ratio of droplets to ions emitted by the sprayer which will then operate essentially as an ion source. It is found in practice that a needle of tip radius in excess of $50 \mu\text{m}$ (compared with $5\text{--}10 \mu\text{m}$ for an ion source) provides the most satisfactory emitter. The prototype sprayer, which is normally mounted on a 70 mm conflat vacuum flange, is shown schematically in Fig. 2. It is designed to spray either horizontally or vertically, though in the vertical mode care must be taken not to overfill the device with consequent flooding of the needle due to hydrostatic pressure.

Also illustrated in Fig. 2 is the electrostatic lens and deflection arrangement used in the preliminary experiments for maskless pattern generation.

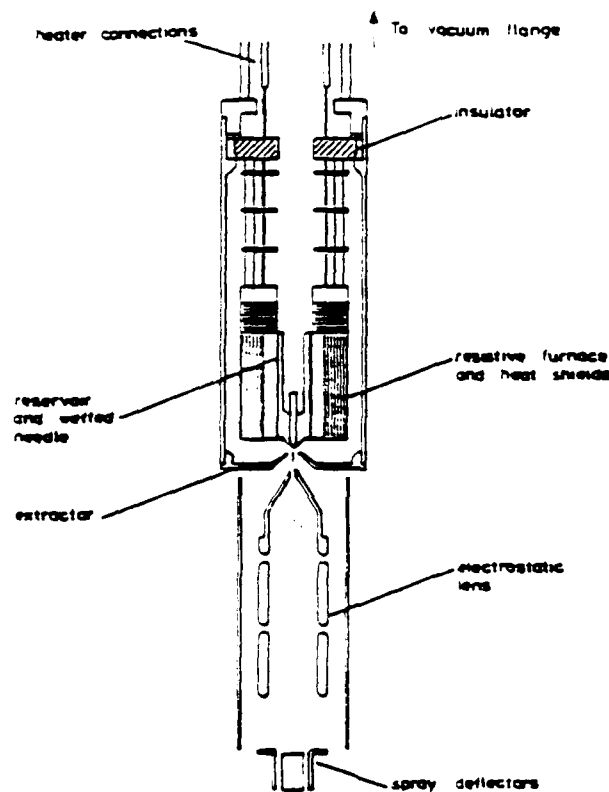


Fig. 2. Schematic diagram of prototype sprayer with focusing lens and deflectors.

3. SPRAYER PERFORMANCE

The onset voltage for emission from a blunt needle FED sprayer is typically about 12 kV. At currents of up to about $150 \mu\text{A}$ the spray is composed chiefly of ions, though there is evidence to suggest the existence of clusters and microdroplets with diameters ranging from a few hundred angstroms to about 1000 \AA . In this mode the

high energy metal ions are used to sputter-clean the surface prior to deposition. At higher currents (corresponding to higher voltages and electric fields), increased field-disruption of the liquid surface occurs and the ratio of droplets to ions in the spray is increased. In this mode, IONCOTE deposits are produced by agglomeration of the energetic droplets which solidify on impact with the substrate. This coating mechanism is confirmed by scanning electron micrographs (SEM) of the coated surface as shown in Fig. 3. The coating mode begins at a current of about 200 μA (see Fig. 3(a)) when the average droplet diameter d is less than 1 μm and the deposition rate R is correspondingly small ($R < 0.2 \mu\text{m cm}^2 \text{ min}^{-1}$). At much higher currents (greater than 700 μA , see Fig. 3(c)), deposition rates are significantly increased ($R > 1 \mu\text{m cm}^2 \text{ min}^{-1}$) but this is achieved at the expense of deposit quality which is far coarser ($d \approx 4 \mu\text{m}$). The optimum coating mode for most applications occurs at currents of about 600 μA ($R \approx 0.5 \mu\text{m cm}^2 \text{ min}^{-1}$, $d \approx 2 \mu\text{m}$), as shown in Fig. 3(b).

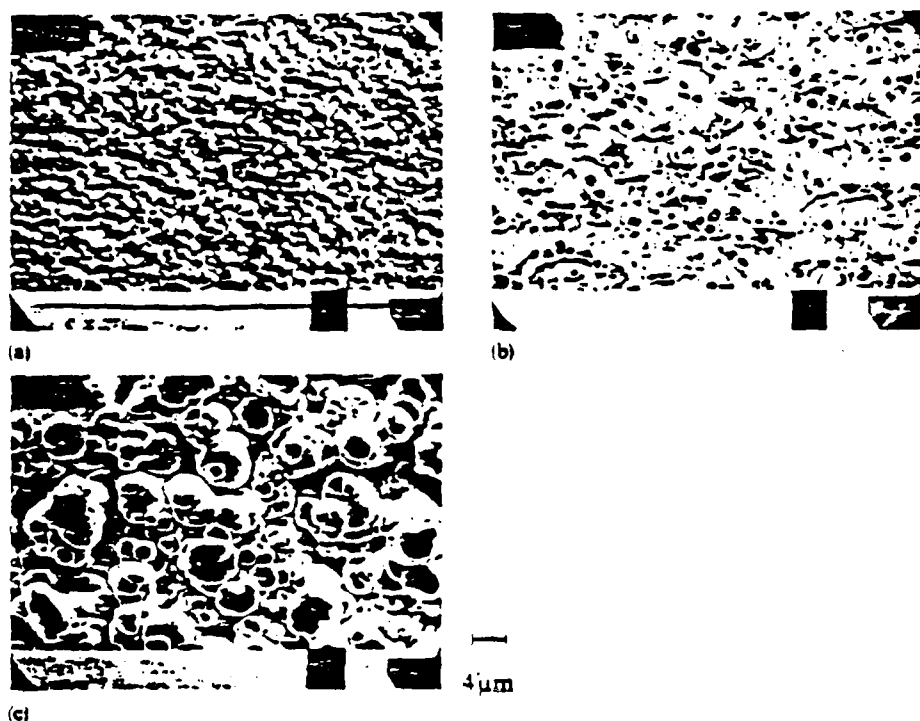


Fig. 3. SEM micrographs of FED coatings showing variation of droplet size with emission current for an extractor diameter of 4.15 mm. (a) Fine deposit: $I = 200 \mu\text{A}$; $V = 14 \text{ kV}$; $R < 0.2 \mu\text{m cm}^2 \text{ min}^{-1}$; $d < 1 \mu\text{m}$. (b) Optimum deposition: $I = 600 \mu\text{A}$; $V = 17 \text{ kV}$; $R \approx 0.5 \mu\text{m cm}^2 \text{ min}^{-1}$; $d \approx 2 \mu\text{m}$. (c) Large droplet deposition: $I \approx 725 \mu\text{A}$; $V = 15.5 \text{ kV}$; $R \approx 1.0 \mu\text{m cm}^2 \text{ min}^{-1}$; $d \approx 4 \mu\text{m}$.

These variations are further illustrated by the metallurgical sections of IONCOTE deposits as shown in Fig. 4 (in this case for films of aluminium of thickness in excess of 10 μm). Figure 4(a) shows porosity in the film associated with the presence of large droplets in the coating spray. Figure 4(b) shows the dense film structure obtained by coating in the optimum mode.

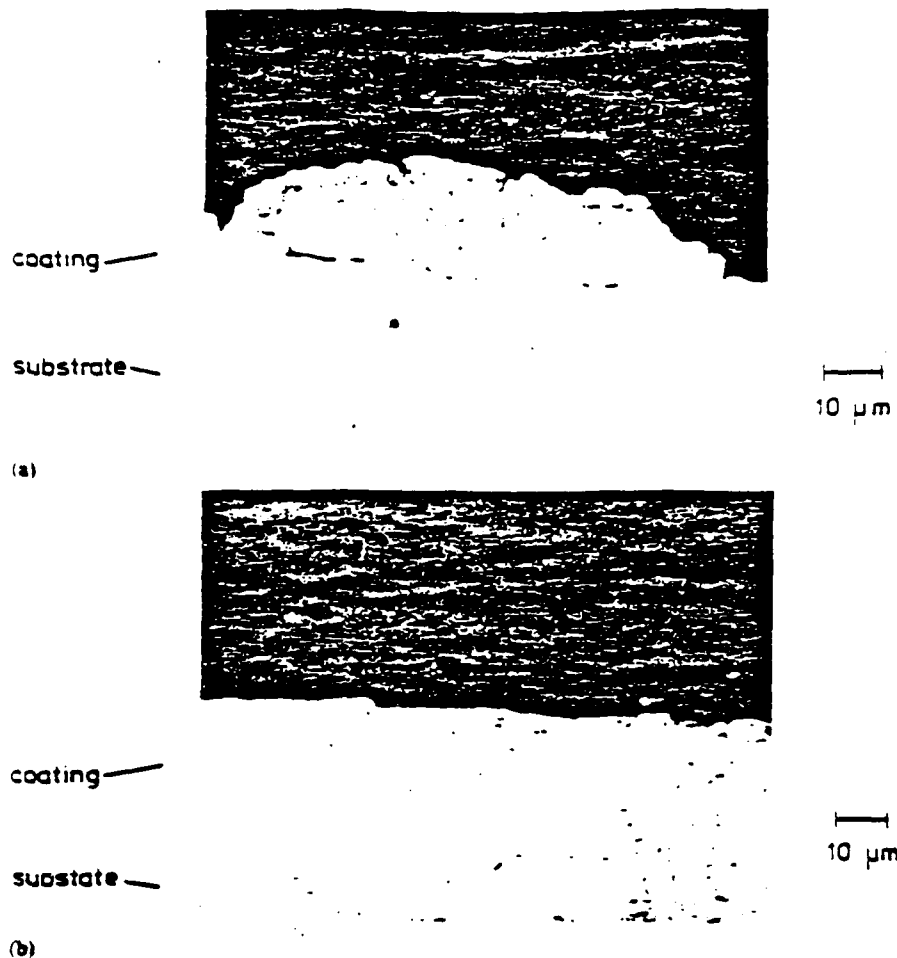


Fig. 4. Metallurgical sections of FED coatings comparing the porous deposit obtained using excessively large droplets at higher currents (a) with the dense structure obtained under optimum deposition conditions (b).

If the coating spray is not focused, the divergent beam of droplets and ions causes a peaked axially symmetrical coating to be produced. Thickness profiles of the coatings formed using a gold sprayer on a substrate placed 48 mm from the emitter were obtained using a Dektak profile recorder. Computer-fitted coating profiles obtained from this data are shown in Fig. 5. The sharply peaked profiles in Figs. 5(a), 5(b) and 5(c) refer to the three deposits shown in Figs. 3(a), 3(b) and 3(c). The effect of ion sputtering during coating is clearly seen in Fig. 5(a), which shows a net erosion of the substrate rather than a coating, at distances greater than 9 mm from the centre of the deposit. This effect is not present in the deposit profiles shown in Figs. 5(b) and 5(c) for which the mass deposition rate due to droplets exceeds the mass loss due to sputtering over the whole of the measured region.

Attempts to measure adhesion of FED coatings of gold on a variety of

substrates including glass have revealed an exceptionally high adherence. Values in excess of $2 \times 10^7 \text{ N m}^{-2}$ have been obtained with failure occurring in the bulk substrate or at the test stub-gold interface with no failure of the gold-to-substrate bond. Recent thermal compression bonds using silver deposits have achieved strengths of up to 10^8 N m^{-2} .

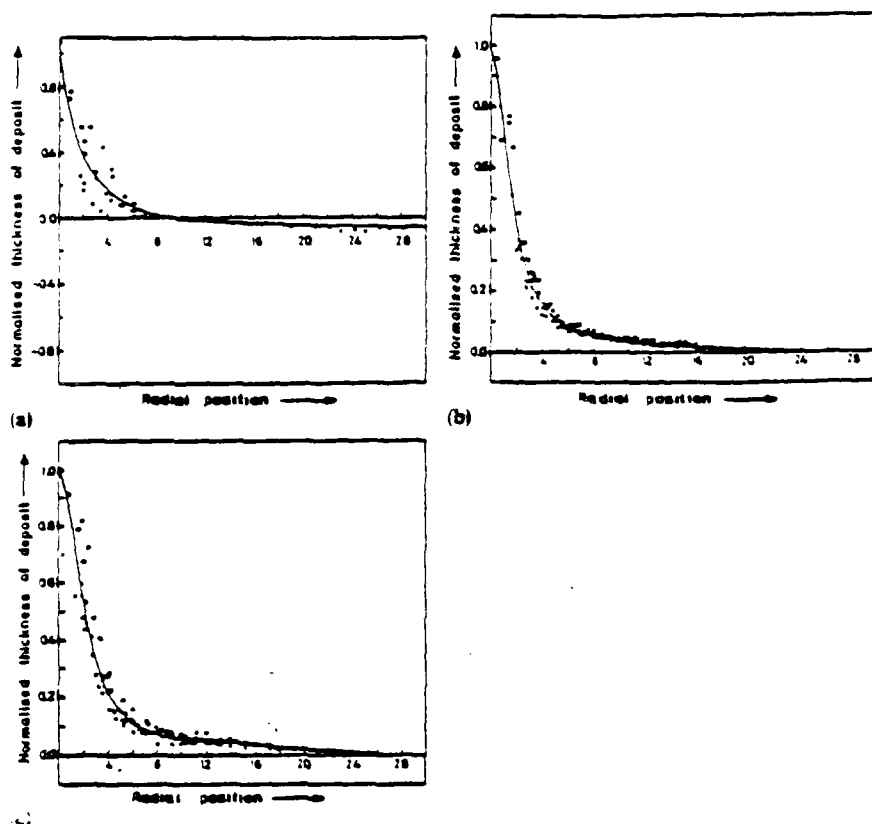


Fig. 5. Measured deposit profiles as functions of sprayer current corresponding to deposits as in Fig. 3: (a) small droplet deposition (note sputtering beyond 9 mm radius); (b) optimum deposition; (c) large droplet deposition.

Electrostatic focusing of the coating spray down to spot sizes of a few millimetres has been achieved. Figure 6 shows the focused spray from an FED sprayer as viewed telescopically using an image-intensifying camera. The sequence 1-4 shows the focused spray being deflected from a central position to strike the substrate at a point outside the circle of light cast by the furnace of the sprayer. This mode of operation is likely to be of particular interest for the maskless generation of hybrid microcircuit interconnects.

4. DISCUSSION AND CONCLUSIONS

As a highly directional vacuum-coating technique, the FED or IONCOTE

process is similar in some respects to ionized cluster beam deposition as developed by Takagi *et al.*⁶ However, the coating particles are much larger in the case of FED and migration effects are subsequently reduced. It is possible to produce amorphous deposits by FED as demonstrated for a Ni-Nb alloy⁷ and, more recently, for silicon⁸.

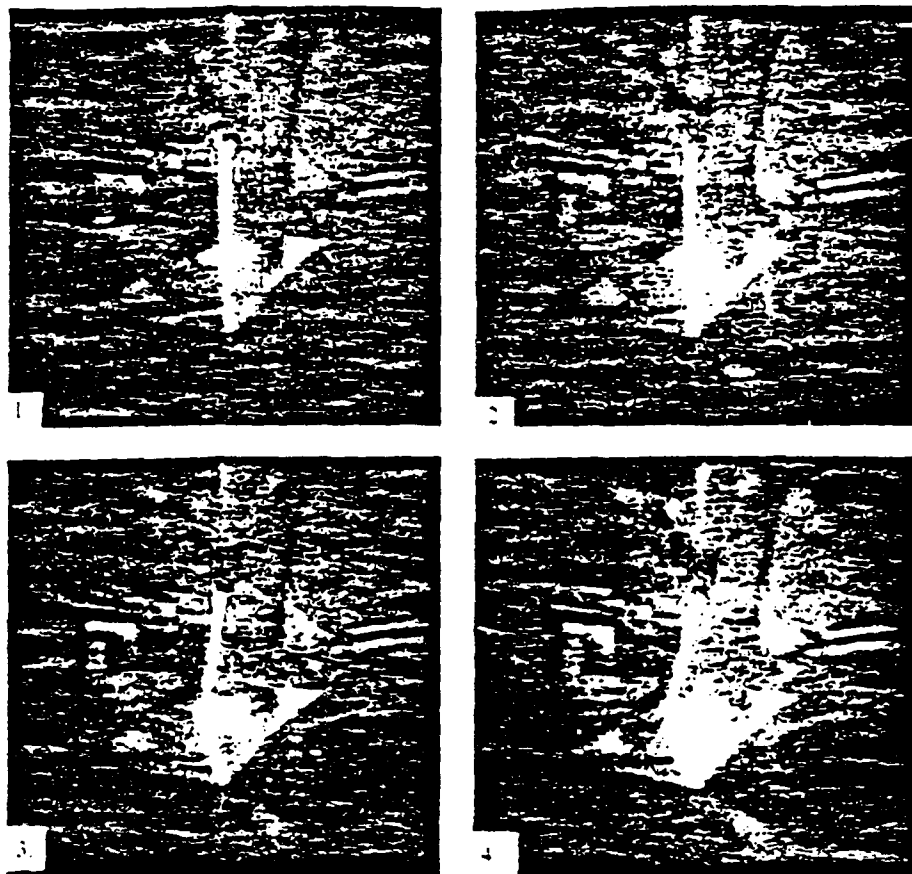


Fig. 6. Photographic sequence showing electrical deflection and focusing of the coating spray.

The energetic metal ions in the spray are felt to be the key to the high adherence which can be achieved. In addition to their sputter-cleaning effect, the ions will undergo shallow implantation into the substrate. This effect, in addition to sputter mixing of substrate and coating, probably plays a significant role in establishing the observed high interfacial bonding.

Our increased understanding of the hydrodynamics of sprayer operation has resulted in much closer control of the droplet size and coating rates. For most applications, maximum droplet diameters of about 2 μm give the optimum results. However, there will probably be growing interest in the low current mode which has hitherto been used solely for ion-sputter preconditioning of substrates prior to

deposition. The tiny clusters and microdroplets present in this mode might well be used to generate microcircuit conductor patterns to submicron tolerances. Maskless pattern generation appears to be the most important potential application of FED technology. Though much work on focusing and deflection of the spray remains to be done, the successful preliminary experiments reported here for the first time indicate the considerable promise of the FED process.

ACKNOWLEDGMENTS

The prototype sprayer and lens system described in this paper is being developed as a commercial module in collaboration with Dubilier Scientific Ltd., Abingdon, Oxfordshire, Gt. Britain.

The authors are grateful to Mr. D. J. McMillan and Mr. G. R. Horsell of Culham Laboratory for experimental and technical assistance. Mr. I. L. Robertson is thanked for his advice on computational matters.

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